Liquid Chlorine

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Chlorine Exposure Limits
Chlorine Facts

Liquid Chlorine (LCl_2)

Background

Liquid chlorine inside an open glass vial

The periodic table contains eleven elements which are in the gas phase under **standard conditions**, namely hydrogen, nitrogen, oxygen, fluorine, chlorine, and the six **noble gases**. Of these elements, chlorine has the highest **boiling point** of -34.0 °C (239.1 K, -29.3 °F). Although this may seem exceedingly cold compared with a comfortable room temperature, it is easily achievable in a laboratory (or home) setting with a few common supplies. Additionally, in some of the coldest regions on Earth (such as **Antarctica**), the outside temperature regularly drops below chlorine's boiling point. It is therefore relatively easy to prepare liquid chlorine as is demonstrated below.

Michael Faraday was a brilliant scientist who made numerous important contributions to the fields of physics and chemistry. One of the many things he is remembered for is being the first person to liquefy chlorine gas. In 1823 Faraday discovered that putting chlorine under pressure and cooling it in an ice bath caused the gas to condense into a yellow liquid.

Pressurizing chlorine requires a pump immune to the highly corrosive nature of the gas. Additionally, putting chlorine gas under pressure creates the risk of springing a leak in, or possibly a catastrophic failure of, the containment vessel, thereby allowing chlorine to escape into the surroundings where other people or equipment may be located. For these and other reasons it is much more desirable to approach the task of liquefying chlorine gas, on the small scale, using an alternative method than that employed by Michael Faraday, one which was not available in 1823.

⚠Warning⚠

Chlorine is toxic and can cause severe respiratory damage and, if inhaled in sufficient quantities, even death. Take great care to avoid breathing chlorine gas. In case of inhalation, retreat to an area with fresh air immediately. Consult the **MSDS**, and other reliable sources, to determine the appropriate medical attention required for various levels and paths of exposure to dangerous substances.

Chlorine gas, and liquid chlorine, are highly corrosive and may act as an oxidizing agent to many organic and metallic materials.

Dry ice and especially liquids cooled using dry ice pose a significant safety risk. Do not allow these substances to touch living tissue (for example, skin) for any significant period of time as they will quickly cool the tissue to dangerously low temperatures and can result in frostbite. Always handle with thermally-insulting, **non-absorbent** gloves.

A list of applicable MSDS pages are provided in the 'external links' section on the left.

Only experienced persons possessing the proper equipment and who are knowledgeable of the material's properties and the recommended safety procedures should attempt this experiment. It is only advisable to perform this experiment inside a well-maintained fume hood or glove box in order to protect oneself from the corrosive and toxic effects of liquid and gaseous chlorine. The danger may be further minimized by only producing chlorine gas, and thus liquid chlorine, in small quantities. Proceed with Extreme Caution and at One's Own risk.

Dry ice (carbon dioxide in the solid phase) **sublimes** at a temperature of -78.5 °C. One could use dry ice directly to condense chlorine gas, however a more efficient method uses the dry ice to cool a liquid down to the same sublimation temperature, which is then used to cool the chlorine. The chilled liquid possesses the advantage that it will conform to the external shape of the chlorine condenser, giving it a greater contact area over solid chunks of dry ice which will not pack perfectly around a rigid container. The greater contact area between the “hot” (chlorine) and “cold” (ice-bath) reservoirs will allow for faster heat transfer, and thus more efficient cooling and condensation of the chlorine. The trick is choosing a liquid that will not freeze at such a low temperature. For this purpose “denatured alcohol” was chosen to act as the cooling bath fluid to be used in conjunction with dry ice, for reasons explained below.

Denatured alcohol is a somewhat generic term that applies to a mixture of different types of alcohol which is not intended for human consumption (in fact, drinking it may lead to serious illness or death). Generally denatured alcohol contains ethanol as well as additives that make it poisonous, such as methanol and isopropanol, but the precise makeup and proportions can vary between products. Denatured alcohol is a commonly used solvent sold in hardware

stores for paining and other purposes. Since the alcohols which are mixed to make up denatured alcohol have very low freezing points (ethanol freezes at about -114 °C, methanol at -97 °C), it will not freeze when dry ice is added. Additionally, denatured alcohol is relatively inert to many materials (plastics, glass) which might be used to contain the cooling-bath as well as the flask used to condense the chlorine, therefore one does not need to worry about the container dissolving as one might with other organic solvents such as acetone or toluene. This property will become important later as a Styrofoam container (desirable for its insulating properties) was chosen to hold the cooling bath and would not have been able to withstand the effects of harsher organic solvents.

Apparatus Setup

The apparatus used to generate, dry, and then liquefy, chlorine is depicted in the diagram below. The complete setup is divided into 3 stages: the chlorine generator (left), bubbler (center), and condenser (right). Each of the three stages are discussed below.

Enlarge Image: With Labels --- Without Labels

Note: Ideally, all of the ground glass joints in the above apparatus should be secured with Keck clamps (not shown) in order to ensure they remain tightly sealed during operation. As chlorine gas is generated the pressure inside the flasks will increase. The increase in pressure may accidentally loosen, or break, the seal between one or more pieces of glassware, thereby leaking chlorine into the surroundings and disrupting the proper flow of gas into the condenser. The clamps hold the joints securely in place and prevent this occurrence.

Chlorine Generator

A flat bottom flask containing solid granules of calcium hypochlorite ($Ca(OCl)_2$) was attached, via a 3-neck Claisen adapter (only 2 necks are depicted in the diagram since the 3rd neck is unused), to an addition funnel containing hydrochloric acid (HCl) and to a barbed hose adapter. This assembly is identical to that shown here, where the addition funnel extends up beyond the top of the image (Note: this image is taken from another project, but the arrangement is the same as that used here).

Once properly assembled, this arrangement allows for the controlled addition of hydrochloric acid to the flask containing $Ca(OCl)_2$. When mixed, HCl and $Ca(OCl)_2$ react to produce chlorine gas, as described by the chemical equation shown below. Since the container is completely sealed except for the opening in the barbed hose adapter, the chlorine is directed out of the flask and through the connected tubing. Such a setup is advantageous when generating chlorine gas since the reaction is completely contained inside the glassware and the operator is never directly exposed to the chlorine or its precursor chemicals.

$$Ca(OCl)_2 + 4\,HCl \longrightarrow CaCl_2 + 2\,H_2O + 2\,Cl_2$$

Alternative methods exist to generate chlorine gas and one will find a more detailed discussion on this topic in the related article, "Chlorine Gas Production". For the purposes of liquefying chlorine, the precise method and apparatus used to generate the gas do not matter a great deal, the above method is only one suggestion. Any chlorine gas source may be used in place of the generating apparatus depicted on the left side of the above sketch whose output is then fed into the next stage (the bubbler).

Bubbler

Water vapor is mixed in with the chlorine during production since the gas is generated in a very water-rich environment (hydrochloric acid contains a great deal of water, in addition to the water that is formed alongside chlorine in the chemical reaction). Thus, the as-produced chlorine gas is considered "wet". It is desirable to "dry" the gas prior to liquefying since this increases the purity of the final product as well as makes the liquid chlorine less corrosive to many materials.

In order to remove this water, the chlorine gas is directed into a sulfuric acid bubbler following production. Concentrated sulfuric acid is a powerful desiccant and readily absorbs water from its surroundings. As the chlorine is bubbled through the acid, water vapor is removed from the gas and the chlorine which exits the bubbler is left very dry (i.e it contains very little moisture). One could also use an alternative dessicate, such as calcium chloride packed inside a long sealed tube through which the chlorine gas is passed. Once the chlorine is dry it is then ready for condensation into a liquid.

In the diagram, chlorine gas is fed into the bubbler through the lid and down a tube to the bottom of the container. Upon exiting the tube, the chlorine gas bubbles up to the top of the liquid, in the process contacting the sulfuric acid and loosing some of the water vapor mixed in with the gas. The dry chlorine gas accumulates at the top of the bubbler and exists through a tube connected to the outlet. The dehydration process is fastest when the contacting surface area

between the wet gas and the dehydrating agent is maximized, thus smaller bubbles are more efficient at drying the gas due to the larger surface area to volume ratio.

Chlorine Condenser

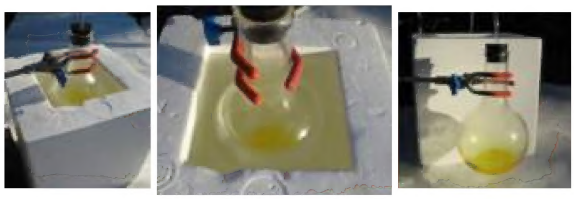
A borosilicate glass (Pyrex) flat bottom flask was used as a condenser in this experiment. The Pyrex flask was chosen since it is chemically inert, can withstand the thermal shocks involved in condensing chlorine, and allows for high thermal contact areas between the chlorine and cooling bath.

The flask was immersed in a dry ice and alcohol bath in order to keep its wall's chilled to a temperature of approximately -78 °C throughout the course of the experiment. Dried chlorine gas was directed into the flask though a loose-fitting rubber stopper with a single hole drilled in its center, through which a chlorine-carrying tube was inserted. Chlorine gas which enters the flask condenses on its walls and accumulates at the bottom of the flask where the temperature is maintained at -78 °C by the surrounding dry ice and alcohol bath. If the rate at which chlorine gas enters the flask exceeds the rate of condensation, the pressure inside the flask will rise and push open the loose-fitting stopper, venting the flask in a controlled manner and avoiding undesired pressure buildup.

The liquid chlorine which condenses is cooled to the temperature of the dry ice bath, which is well below chlorine's boiling point. At this temperature, though, chlorine still has a vapor pressure of approximately 15 kPa (about 15% of standard atmospheric pressure). As a comparison, ordinary pure water has a vapor pressure of 15 kPa at a temperature of about 54 °C (130 °F). Like hot water, liquid chlorine will evaporate as some of its molecules will transition into the gas phase (even though it is below its boiling point). Just because the chlorine is a cold liquid does not mean that there is no chlorine gas around, thus one should continue take care not to inhale the fumes.

A Styrofoam container was used to contain the dry ice and alcohol cooling-bath which surrounded the chlorine condenser. Styrofoam is an excellent thermal insulator which reduced the flow of heat into the cooling bath and helped maintain its low temperature longer (reduce the amount of dry ice needed to keep the liquid cold for long periods of time).

Pictures of the actual chlorine condensing setup may be seen below.



Below, one may see pictures of a small amount of liquid chlorine contained within a glass vial.

It is interesting to note the effect on the polyvinyl plastic tubing used in this experiment due to the many hours of constant exposure to chlorine gas. The tubing shown below was initially perfectly clear, as can be seen in comparison with a used hose in the second photograph. After many hours of chlorine exposure, the hose turned a bright, opaque, yellow color on its inside surface.

Liquid Chlorine Videos

In the below video one can see as the chlorine condenser is removed from the cooling-bath and shaken, causing the yellow-colored liquid chlorine contained within the flask the move and swirl.

Video

YouTube

Chlorine can act as an oxidizing agent on many organic materials. As a means to test the oxidizing power of liquid chlorine, small amounts of the liquefied element were poured onto items of interest such as a piece of dyed fabric and (as shown below) a piece of paper with a picture printed on it. As mentioned above, Michael Faraday was the first person to liquefy chlorine. For this reason, and out of great respect for his achievements, an ink-jet printed portrait of Michael Faraday was chosen to test the oxidizing effects on liquid chlorine. Only dry (containing little to no water) liquid chlorine was used for these tests.

Video

YouTube

As the liquid chlorine is poured out onto the paper nothing particularly spectacular is observed to occur; the liquid simplyflows to cover and soak into the fibers of the paper. Afterward, the chlorine is allowed to warm up and eventually boil away over the course of several minutes. Once the paper was dry, and all the chlorine vaporized, the paper was compared with an identical copy that had not been soaked in liquid chlorine, as seen below.

Above: original, un-soaked, picture (left), chlorine-soaked picture (right)

Notice the difference in color between the original and liquid chlorine-soaked pictures. The chlorine-soaked picture (right) has a green tint, which would seem to indicate selective oxidization / bleaching of the ink used to print the image.

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